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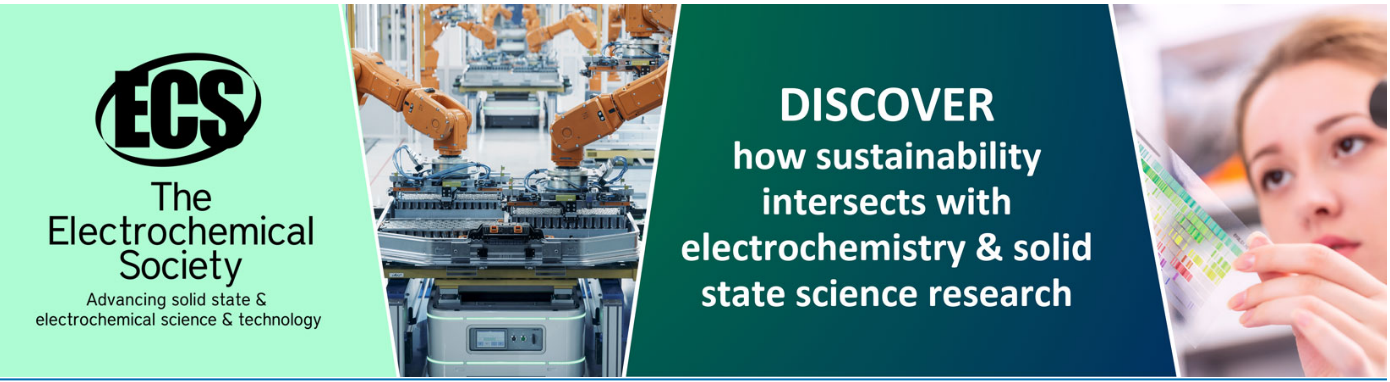

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Modification and Applications of Hydrophilic Polypropylene Membrane

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Abstract. Polypropylene (PP) is one of the most important polymers for microporous membrane due to its high void volume, well-controlled porosity, high thermal and chemical stability, and low cost. However, the hydrophobicity of PP becomes a limitation to broaden its applications. Furthermore, membrane fouling occurs more seriously on hydrophobic membranes than hydrophilic ones. To solve this problem, surface modifications have been developed to enhance PP membrane hydrophilicity without changing its bulk properties. Graft polymerization and plasma treatment are the most popular techniques for surface hydrophilization. Some studies showed that highly hydrophilic PP membranes with water contact angle less than 20° could be obtained by plasma treatment and graft polymerization. Furthermore, during plasma treatment, polar groups were formed on the PP membrane surface thus increased water uptake. To bring brief explanation on various research trends for PP modification, this paper provides a review of surface hydrophilization of microporous PP membrane, including plasma treatment and graft polymerization. The effects of surface modification on PP membrane performance such as porosity, water contact angle, and water flux are also discussed. In addition, the applications of modified PP membrane are presented as well.

1. Introduction

Membranes have been used for various separation processes, such as water and wastewater treatment, fuel cells, artificial organs, and industrial filtration. For each applications, the separation performances are determined by membrane characteristic (Ulbricht, 2006; Sykes *et al.*, 1982). For a polymer membrane, the performances depend much on the chemistry of the membrane material, the structures and properties of the porous layer, and the microstructures and surface properties of the top layer (Wan *et al.*, 2009).

Many of the polymers which are used for commercially available synthetic membranes are hydrophobic polymers. (Chung, 1998). Polypropylene (PP) is one of the polymer that has been used extensively as membrane material due to its chemical stability, low cost, good mechanical properties, and being readily available (Bhat *et al.*, 2003; Shahidi *et al.*, 2007; Yu *et al.*, 2007a; Yan *et al.*, 2008; Yang *et al.*, 2005; Meng *et al.*, 2014; Xu *et al.*, 2013; Feng *et al.*, 2011; Himma *et al.*, 2016b). PP membranes are used for a variety of industrial applications, such as sterilization of beverage and pharmaceutics, wastewater treatment, ultrapure water in the semiconductor industry, desalination of sea water, electrode separation, etc. (Bae *et al.*, 2001; Mulder, 1996). However, the application of PP membrane is restricted by its low energy surface and relatively high hydrophobicity that lead to membrane fouling. Therefore, surface modification is emerged to obtain hydrophilic PP membranes and increase antifouling characteristics.

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Surface modification of polymer membranes has emerged to develop membranes separation performance and stability. Generally, the aim of membrane surface modification is to minimize fouling, modulate hydrophilicity/hydrophobicity, or enhance biocompatibility (Wan *et al.*, 2009; Bae *et al.*, 2001; Corn *et al.*, 1991; Guo and Ulbricht, 2010). Various techniques are available for surface modification of PP membranes, such as chemical, flame, corona, UV irradiation, and plasma treatment, etc.(Sykes *et al.*, 1982; Briggs *et al.*, 1979; Bhat and Upadhyay, 2002; Lanauze and Myers, 1990; Singh *et al.*, 2008; Lipnizki *et al.*, 1999). Among the various surface modification techniques, plasma treatment and graft polymerization are regarded as the most attractive methods for hydrophilization of PP membranes.

Hydrophilicity refers essentially to hydrogen bonding with water molecules. Therefore, having a high percentage of oxygen, either in the main chain or in pendant groups, contributes to the hydrophilicity of a material (Zou *et al.*, 2011). A thin layer of bound water exists at the surface of hydrophilic membranes. This layer prevent or reduce the undesirable adsorption or adhesion of foulants at the membrane surface (Kochkodan and Sharma, 2012). Thus, the anti-fouling property of membrane can be promoted by improving its hydrophilicity (Wenten, 1995).

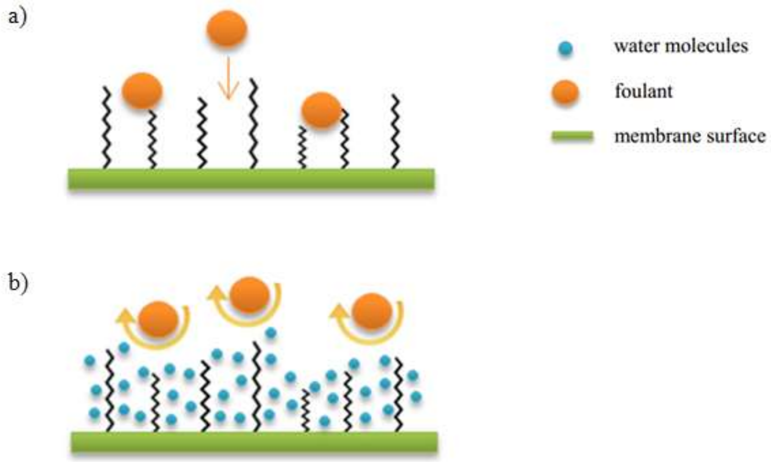

Figure 1. Interaction between foulant and membrane surface of: a) unmodified PP membrane and b) hydrophilic modified PP membrane, adapted from (Zhang *et al.*, 2016)

Some reviews relating to the modification strategies of PP membrane have been published. However, a review of surface hydrophilization of PP membrane has not been reviewed elsewhere. To provide an insight for the development of hydrophilic PP membrane in the future, this paper provides a review of surface hydrophilization of microporous PP membrane, including plasma treatment and graft polymerization. The effects of surface modification on PP membrane performance such as porosity, water contact angle, and water flux are also discussed. In addition, the applications of modified PP membrane are presented as well.

2. Plasma Treatment

Plasma is a quasi-neutral gas consists of electrons and ions as well as neutrals, atomic, and molecular species that exhibit a collective behavior in the presence of an electromagnetic field (Gomathi *et al.*, 2008; Ciszewski *et al.*, 2006). Plasma treatment processes have been extensively studied to modify the surface of PP membranes without affecting the bulk properties. The major effects from plasma treatment on the polymer surfaces are cleaning of organic contamination, micro-etching, cross-linking, inducing hydrophilic modification and improving adhesive properties of polymeric substrates (Bhat and Upadhyay, 2002; Liston, 1989; Hollahan *et al.*, 1969; Boyd *et al.*, 1997; Owens, 1975).

In general, the surface energy of untreated PP membranes is too small for good adhesion. For optimum adhesion, the surface energy of the membranes should be larger than that of the material to be bonded with. Similarly, for effective wetting by a liquid, the surface energy of the polymer membranes should exceed the surface tension of the liquid (Rajesh and Mark, 2003). By using plasma treatment, the overall surface energy of PP membrane can be increased.

Surface modification of PP membranes by plasma treatment can be achieved using gases such as air, oxygen, nitrogen, ammonia, argon, etc. (Yu *et al.*, 2007a; Liston, 1989; Boyd *et al.*, 1997; Rajesh and Mark, 2003; Yu *et al.*, 2008; Slepička *et al.*, 2010). Gas molecules in a plasma reactor are activated by the collision with electrons, positive ions, and metastable species that subsequently attack on PP membrane surface (Yasuda and Wang, 1985). Oxides and nitrides thus formed lead to accomplishment of surface hydrophilic modification. Therefore, the gases that used for the plasma treatment determine the hydrophilicity (Shahidi *et al.*, 2007; Bhat and Upadhyay, 2002; Chaivan *et al.*, 2005).

Table 1. Modified membrane characteristics after 20 minutes of plasma treatment

Plasma treatment	Average pore size (μm)	Porosity (%)	Water contact angle	Relative water flux, J/Jo	Ref.
N_2	-	-	86°	1.70	(Yu *et al.*, 2007a)
NH_3	0.10 ± 0.02	44.4 ± 2.7	58°	1.19	(Yan *et al.*, 2008)
Allylamine	-	-	70°	-	
Acrylic acid	-	-	42°	-	(Bae *et al.*, 2001)
H_2O	-	-	32°	-	
Air	0.09 ± 0.02	-	35°	1.30	(Yu *et al.*, 2008)
O_2	-	-	15°	-	(Yun *et al.*, 2004)
CO_2	0.10 ± 0.02	35.4 ± 1.8	76°	1.21	(Yu *et al.*, 2005)
CH_4-O_2	-	-	8°	-	(Tsai *et al.*, 2011)

The wetting properties are significantly affected according to the types of plasma treatment species. Water contact angle (WCA) measurements have been commonly used to characterize the hydrophilicity of the membrane surface. As shown in Table 1, plasma treatment can modified PP membranes to be hydrophilic ones. WCA hysteresis is happened due to the change of the surface roughness and hydrophilic characteristic. The WCA on a rough surface is smaller than that done on a smooth surface (Bae *et al.*, 2001). However, Yu et al. (Yu *et al.*, 2007a) and Yan et al. (Yan *et al.*, 2008) showed that the WCA increased with the increase of storage time. It indicates that hydrophilicity gained by plasma modification is not stable. The effect of plasma treatment can get lower and even disappear due to mobility of surface functionalities.

Beside decrease WCA, plasma treatment usually generates the presence of polar groups on the membrane surface. The presence of polar groups on a PP membrane surface also enhances the hydrophilic property of that PP membrane surface, thus easing water molecules to get through the membrane, which increases water uptake (Himma *et al.*, 2016a). Previous study (Hansen and Schonhorn, 1966) also showed that crosslinking initiated by activated species of inert gas as one of the reasons for improving surface cohesive bonding. Furthermore, the result from another study (Chung, 1991) indicated that bond-ability improves by intimate molecular contact and by inducing attractive force between the substrates. Molecular contact can be improved by inducing surface crosslinking, whereas attractive force between the substrates can be improved by surface hydrophilic modification.

Hydrophilic modification of the membrane surface by plasma polymerization also showed the effect of reducing organic absorption. Although the plasma polymerization treatment caused a reduction in initial membrane permeability, overall performance of the membranes showed almost constant fluxes for the whole experiment. This reduction of the initial flux of the treated membrane could respond to the small voids on the membrane surface that are partially filled with plasma polymer by the coating process (Zou *et al.*, 2011). Furthermore, the improvement of hydrophilicity varies according to plasma treatment time. Membranes with a shorter treatment time have better filtration results than those with a longer treatment time.

3. Graft Polymerization

Surface modification by graft polymerization is done by tethering polymer chains onto polymer membrane. It is a useful method for the development of membrane surface properties and bulky strength (Meng *et al.*, 2014; Kato *et al.*, 2003; Uyama *et al.*, 1998). Graft polymerization provides existing polymer membranes with new functionalities such as hydrophilicity, adhesion, biocompatibility, conductivity, anti-fogging, and anti-fouling. Monomer react with a membrane surface that has been activated, then another initiator is immobilized onto the membrane surface, where it will then be activated to initiate the polymerization process with monomer (Himma *et al.*, 2016a). Graft polymerization is very effective to modify PP membrane with stable hydrophilicity.

A variety of methods for graft polymerization onto PP membrane surfaces has been reported. The methods include chemical graft polymerization and grafting with the use of high energy radiation or oxidizing agents (Uyama *et al.*, 1998). Functional groups are first introduced to the polymeric substrate by UV irradiation, glow-discharge, or ozone and then are decomposed and initiated for polymerization. For materials with reactive groups on the surface, the desirable polymer can be easily tethered on through the free radical graft polymerization of a monomer or the chemical reaction of a polymer with functional end groups. Meanwhile, materials without reactive groups on the surface need plasma, gamma-ray irradiation and UV photo induced graft polymerizations (Liu *et al.*, 2004).

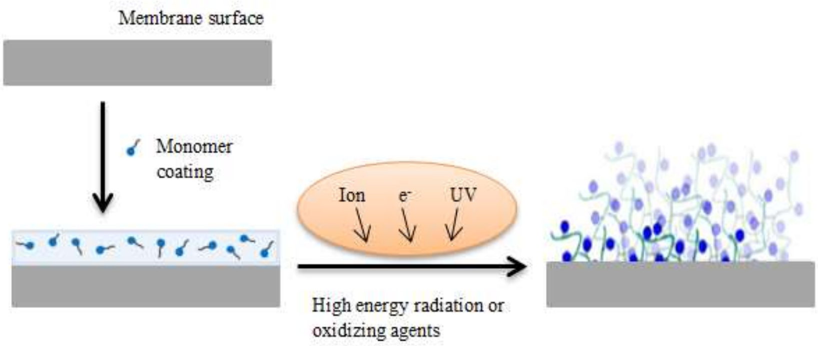

Figure 2. Illustration of PP membrane hydrophilization by graft polymerization, adapted from (Venault *et al.*, 2013)

Based on its grafting methods, graft polymerization can be divided into three types:

- Chemical compound induced. In chemical compound induced, a radical for the polymerization process is formed on the membrane surface through reaction with chemical compounds. One of the most attractive techniques for oxidizing a substrate surface is ozonation (Uyama *et al.*, 1998).
- Plasma induced. In plasma induced method, plasma is used to provide active sites either on the membrane surface or other initiators that can initiate the polymerization process. Plasma treatment is firstly performed to generate radicals on the membrane surface, which then the radicals initiates graft polymerization of monomers.
- Irradiation induced. Irradiation can be utilized to perform graft polymerization on a PP membrane surface by using ultra-violet (UV) and γ-ray as the initiators. PP membranes are first activated by thermal initiators or high energy irradiation, then followed by chemical grafting of hydrophilic polymer chains (Xu *et al.*, 2013). UV irradiation has been widely used for membrane preparation or modification due to its ability to induce cross-linking in the polymer matrix, thus the membrane selectivity can be improved.

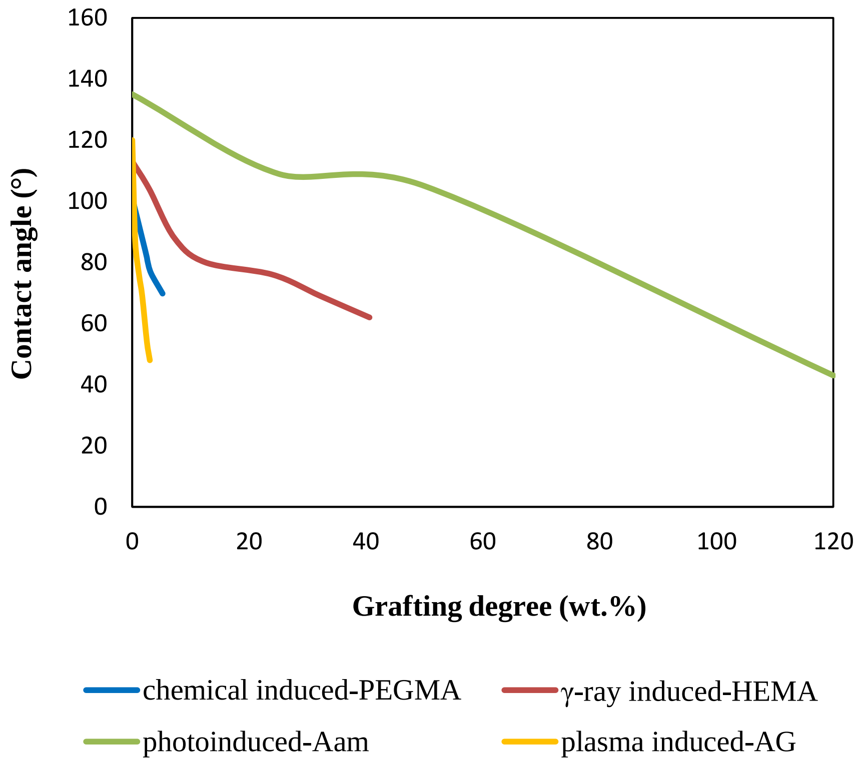

Figure 3. Effect of grafting degree on water contact angle for different methods and polymers (PEGMA: Poly-(ethylene glycol) methacrylate, HEMA: Hydroxyethyl methacrylate, Aam: Acrylamide, and AG: Allyl glucoside), adapted from (Jin *et al.*, 2012; Shim *et al.*, 2001; Yu *et al.*, 2007b; Nie *et al.*, 2003)

The existence of hydrophilic chains on the membrane surface facilitated the reduction of interfacial tension with water. As the result, WCA are drop after modified by graft polymerization. Some literature showed that WCA below 20° can be achieved by graft polymerization (Zou *et al.*, 2011; Zhao *et al.*, 2011; Yang *et al.*, 2010; Zhao *et al.*, 2010; Yang *et al.*, 2009). Furthermore, characteristics of modified PP membrane mainly depend on the grafting degree. The higher degree of graft polymerization provides a strong driving force to reduce the interfacial free energy by migrating and reorienting of the hydrophilic monomers (Jin *et al.*, 2012). As the result, higher grafting degree with a large coverage surface decreases the WCA (as shown in Figure 3).

The antifouling resistance of the modified membranes is also improved due to an increase in their hydrophilicity. Therefore, through surface grafting modification, improvement in the hydrophilicity of PP membrane may bring the increase of water permeation flux due to the balance between the pore-covering effect of the grafted chains and the increase of hydrophilicity. However, the water flux remain almost unchanged when the grafting degree is low, but the negative effect of pore-covering soon become the major factor with the increase of the grafting degree, causing the water flux dropping severely (Dai *et al.*, 2008). Thus, the precise design of a graft layer and well-controlled graft architecture is a very important issue to minimize the membrane fouling.

4. Applications

4.1 Water and wastewater treatment

With the increasing shortage of clean water sources worldwide, the development of advanced water treatment technologies of low-cost and high efficiency is urgent. There are many areas in which membrane water treatment can play an important role, such as drinking water treatment, brackish and seawater desalination and wastewater treatment and reuse (Yin and Deng, 2015). PP membranes have found potentially important applications in water treatment. However, its hydrophobicity and fouling is the major obstacle in the application. Surface modification of PP membrane is then undoubtedly necessary to avoid premature membrane deterioration due to fouling.

One of the most thorough applications studied is the membrane bioreactor (MBR), since fouling is the major concern of MBR (Le-Clech *et al.*, 2006). Previous study showed that the performance of modified hydrophilic PP membrane in MBR is enhanced as indicated by a decrease in fouling tendency (Chung and Lee, 1997), thus promoting it to address the fouling issues during MBR operation. When directly applied in MBR, this efficacy is proven by lower flux reduction and higher flux recovery.

4.2 Biotechnology

The choice of polymer membranes for various biotechnology applications depends on their surface properties. Specific surface properties like hydrophobicity, chemical structures, roughness, conductivity, etc. can be modified to meet the specific requirements (Gomathi *et al.*, 2008). At the most cases, PP membranes are not applicable for biotechnology applications due to the specificity of bio-fluids and biomolecules, which are highly environmentally sensitive. Biomolecules (e.g. enzyme) tend to irreversibly adsorb on the hydrophobic membrane surface. The adsorption causes membrane fouling and induces changes of the structures and functions of the biomolecules (Wan *et al.*, 2009). Therefore, surface modification of PP membrane is necessary to improve biocompatibility of membrane surfaces. Modification of PP membrane surfaces by plasma treatment can improve wettability, oxidize the surface, and enhance cell growth and adhesion (Gomathi *et al.*, 2008).

4.3 Medical

Most biomedical materials are used in constant contact with living systems, such as blood, cells, and tissues. Since the material surface can undergo unfavourable biological responses when in contact with a recipient, most of the conventional surfaces need to be modified so that the materials can function as designed (Uyama *et al.*, 1998). PP membranes, as one of the most popular membrane materials, have been used in many biomedical separation due to their random network of overlapped fibers, multiple connected pores, high thermal and chemical stability (Zhao *et al.*, 2011; Wang *et al.*, 2009; Zheng *et al.*, 2010). PP membranes have been applied for drug delivery, artificial organs (artificial kidney, liver, pancreas, lung, etc.), tissue regeneration, diagnostic devices, and other blood purification processes (e.g. plasma treatment and cell separation) (Wan *et al.*, 2009).

In a blood purification, hydrophilic property is required to some extent in order to prevent protein adsorption while allowing flux of aqueous solution (Gérard *et al.*, 2011). The same problems are also encountered in a blood oxygenator, where protein adsorption and platelet adhesion contribute in decreasing gas permeability. Bovine serum albumin (BSA) is one of the blood proteins that often used as the model to study the efficacy of a hydrophilized PP membrane in regard to its antifouling property. As PP membranes have been successfully hydrophilized, several promising results can be obtained as indicated in reduction of protein adsorption and increasing both permeate flux and membrane flux recovery (Himma *et al.*, 2016a).

4.4 Battery separator

Separator materials for alkaline batteries have received a great attention due to the desire to develop high energy per unit weight and volume battery packages. Battery separator is usually made from a porous membrane that placed between positive and negative electrodes in order to prevent them from contacting one another that can lead to short circuit. There are several important properties of membrane as a battery separator, such as good electric insulation, minimal electrolyte resistance, mechanically and chemically robust, and easily wetted by electrolyte solution (Arora and Zhang, 2004). Surface modified PP membranes can be used for this application since they fulfill basic requirements of a battery separator, such as presence of spontaneous, uniform and permanent wettable surface in order to accommodate and fully retain the aqueous electrolyte solution (Goel *et al.*, 2009).

5. Conclusion

The application of PP membrane is restricted by its hydrophobicity, thus surface modification have been developed to enhance PP membrane hydrophilicity without changing its bulk properties. Plasma treatment and graft polymerization are the most popular techniques for surface hydrophilization. By using plasma treatment, WCA hysteresis is happened due to the change of the surface roughness and hydrophilic characteristic. Plasma treatment also generates the presence of polar groups on the membrane surface. Meanwhile, some studies showed that highly hydrophilic PP membranes with WCA less than 20° can be obtained by graft polymerization. The value of WCA is decreased with the increase of grafting degree.

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